Isolation and Control of Voids and Void-hillocks during Molecular Beam Epitaxial Growth of HgCdTe

D. Chandra, J. Frazier, T.W. Orent, F. Aqariden, S. Gutzler and H.D. Shih

Focal Plane Array Business
Raytheon Systems Company, P.O. Box 655936, Dallas, Texas 75265

ABSTRACT

Void defects were demonstrated to form away from the substrate-epifilm interface during the molecular beam epitaxial growth of mercury cadmium telluride on cadmium zinc telluride substrates. These were smaller in size compared to voids which nucleated at the substrate-epifilm interface, which were also observed. Once nucleated, voids usually replicated all the way to the surface even if the flux ratios were modified to prevent additional nucleation of voids. Occasionally, void defects which close before reaching the top surface without leaving any perturbations on the surface, have also been observed. The voids which form away from the substrate-epi interface, nucleate on defects, frequently hillocks, introduced into the film already grown, leading to formation of defect complexes. These voids can be smaller than 1 μm and appear almost indistinguishable from unaccompanied simple voids. However, these void-hillock complexes displayed a nest of dislocations decorating these defects, which become apparent upon dislocation etching, whereas unaccompanied simple voids did not. The nests could extend as much as 25 μm from the individual void-hillock complex. The density of dislocations within the nest exceeded 5×10^6 cm^{-2}, whereas the dislocation density outside of the nest could decrease to $< 2 \times 10^5$ cm^{-2}.

INTRODUCTION

During an earlier investigation[1] void defects were demonstrated to form away from the substrate-epifilm interface during the molecular beam epitaxial growth of mercury cadmium telluride on cadmium zinc telluride substrates. These were smaller in size compared to voids, which nucleated at the substrate-epifilm interface, which were also observed. Observations of void nucleation away from the substrate-epifilm interface were related to the respective growth regimes active at the time of the void nucleation. Once nucleated, voids usually replicated all the way to the surface even if the flux ratios were modified to prevent additional nucleation of voids.

During the present investigations, additional details of these smaller voids were studied. It was observed that these voids very often existed as defect complexes, where the additional defect in each complex consisted of hillocks. Furthermore, several different varieties of these voids were observed, with some nucleating very close to the top surface

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Chandra, D. ;
Frazier, J. ;
Orent, T. W. ;
Aqariden, F. ;
Gutzler, S. ;

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and some nucleating deep within the film. Among the latter, in some cases, voids were observed to close before reaching the film surface.

EXPERIMENTAL PROCEDURES

The HgCdTe films were grown on (211)B oriented CdZnTe substrates in a custom MBE system manufactured by DCA Instruments. Some HgCdTe films were also grown in the Riber 2300 MBE system. The films were grown on near lattice matched CdZnTe to reduce or eliminate the misfit dislocation density in the epilayers. The substrates were mostly supplied by Raytheon TI systems. A few substrates delivered by Japan Energy (NIMTEC) were also employed. The substrates were etched in 1 % bromine-methanol solution to remove approximately 5 μm of the surface. Rinse in a methanol bath and drying in nitrogen followed this. The drying was performed immediately after the rinse. Detailed discussion of the MBE growth and processing procedures employed can be obtained elsewhere[1-5].

RESULTS AND DISCUSSIONS

Figures 1and 2 display the types of voids observed in these films. The voids vary both in size and shape. Consistent with earlier observations[1], the smaller voids appear to be less irregular or more circular in cross-section. But, each of the smaller voids appears frequently to be a part of a defect complex, as indicated in Figure 3. The nature of the defect complex appears to be most frequently a void-hillock complex, some evidence of which has also been presented earlier[1]. During the present investigations, relatively unambiguous, though still somewhat indirect, indications were obtained that *all* voids which form away from the substrate-epi interface, nucleate on defects which have *already* formed in the film grown upto that point. These first defects appear to be hillocks under all circumstances.

These observations were made by following a consistent series of investigations based on a characteristic of these defects permitting a systematic depth profiling of these complexes. Every one of these defect complexes appears to be associated, or “decorated” with a nest of dislocations. This becomes apparent upon dislocation etching these complexes, as shown in Figure 4, which displays results of defect etching of films containing two different kinds of void-hillocks. In each case, a strong ‘nest’ of dislocation etch pits appears to be visible around each void-hillock complex. The density of etch pits within the nest exceeds 5 x 10^6 cm^{-2}. The nest extends to ~10 - 20 x the size of the original dimension of the void-hillock complex. The size of the defect appears to increase following defect etching. Hence a 0.5 - 2.0 μm sized void-hillock complexes can impact an area ~8 - 35 μm in diameter, whereas a 2.5 - 5.0 μm sized void-hillock complex can impact an area ~25 - 65 μm in diameter.

Each component of these defect complexes, when they existed unaccompanied with the other, *never* displayed an accompanying dislocation nest. Examples of unaccompanied voids not displaying decorating dislocations are displayed following defect etching in Figure 5, whereas examples of unaccompanied hillocks not displaying decorating dislocations are displayed following defect etching in Figure 6. For comparison, an example of as grown hillocks, prior to defect etching, is displayed in Figure 7. Note that for hillocks, defect etching also "etches away" the hillocks themselves, leaving behind features which are no longer recognizable as hillocks (Figure 6).

All these films were grown at a nominal temperature of 180^0C utilizing separate fluxes of Te, CdTe, and Hg at a growth rate of approximately 2-3 μm/h. Detailed procedures have been provided elsewhere.[1,5] Figures 8, 9 and 10 display microphotographs of cross-sections of three separate MBE films. Figure 8 displays the cross-sectional SEM microphotograph of a large void defect. The void clearly nucleates at the substrate-epifilm interface. The defect was generated at the CdZnTe interface during the first stages of nucleation. Parts of this void have been filled with HgCdTe growth, which could be polycrystalline in structure. In general this void is very similar to voids reported by other workers.[3,4] These voids nucleate at particulates present on the CdZnTe surface immediately prior to growth. Figure 9 displays the cross-sectional SEM microphotograph of a medium sized void defect. This void clearly did not nucleate at the substrate-epifilm interface, but at some point near the mid-point of growth. Hence, presence or absence of particulates on the substrate surface, or the quality of the substrate surface itself, did not appear to have a direct influence on this void formation. Once nucleated, the general behavior of this void defect was similar. It tended to increase in size as the growth progressed and reached its maximum size at the end of the growth run. Figure 10a displays the cross-sectional and three dimensional SEM microphotograph of a small void defect. This void nucleated towards the end of a growth run. Like the previous two types of void defects, it also increased in size as the growth progressed, assuming a funnel shaped form. But since the film growth stopped soon after this void nucleated, the void did not get an opportunity to increase in size significantly. Figure 10b displays the top surface of the same MBE film following slight tilting. The defect displayed in Figure 10a is still visible at the lower left edge of the field of view. But most interestingly, voids of comparable sizes appear to have formed throughout the top surface of the film. It is clear that all these voids must have nucleated approximately at the same stage during MBE growth of this film. This point is far removed from the substrate-epifilm interface, and once again particulates on the substrate surface or the substrate surface quality could not have an impact on growth of these voids. These results indicate that voids can nucleate well away from the substrate-epifilm interface, and voids nucleating only at the substrate-epifilm interface, as shown by other workers[3,4], are not the only types encountered in MBE growth of HgCdTe.

These defects appear to arise from fluctuations in growth conditions. Figure 11 schematically displays the growth regimes existing for MBE growth of HgCdTe. With increasing Hg flux, or with decreasing growth temperature, the growth morphology transitions from voids associated with low dislocation density to the optimal growth window displaying few voids, but still with low dislocation density, to formation of twins (hillocks) and increasing dislocation density. Complex conditions emerge when fluctuations occur during growth to force transition from one regime to another regime. If for example, the growth transitions from Hg rich to Hg deficient, then twins or hillocks will form first, followed by void nucleation. As demonstrated above, voids can nucleate away from the epifilm-substrate interface. The hillocks already formed may act as nucleation sites for these void-defects, inducing void formation on hillocks. These new defects will then appear as void-hillock complexes. The reverse scenario is also plausible, leading also to the formation of void-hillock complexes.

These scenarios are most likely during growth of multi-layer films, where it will be necessary to change relative flux magnitudes and growth temperature to move from the growth of a layer of one composition ($Hg_{1-x1}Cd_{x1}Te$) to a layer of another composition ($Hg_{1-x2}Cd_{x2}Te$). This is schematically depicted in Figure 12. Hence, formation of void-hillock complexes will be more likely to occur during a multi-layer and/or multi-composition growth of HgCdTe by MBE, for example the structures MWIR/LWIR/MWIR, than during growth of a single layer of single composition. All films displayed and examined during this examination were multi-layer films.

Ensuring maintenance of the growth within the same regime during the entire process can result in drastically decreasing the concentration of the void-hillock complex defects. Furthermore, it has been possible to maintain the growth within the window displayed in Figure 11, such that a low void density was achievable without leading to formation of high dislocation density. Figure 13 displays the void density and the dislocation density of a MBE growth run maintained within the same growth regime. The void density has fallen to 230 cm^{-2} and the dislocation density has fallen to 8.1×10^4 cm^{-2}.

SUMMARY

Void defects can nucleate at the substrate-epifilm interface as well as away from the substrate-epifilm interface during MBE growth of HgCdTe. The latter kind of void defects does not appear to be arise from particulates or other defects on the substrate surface. These void defects appear to be smaller in size compared to void defects which appear to nucleate at the substrate-epifilm interface. The smaller void defects were also observed to be complexed with hillocks. These new defects acted as sources of nesting dislocations radiating away from the site of the defects. Elimination of fluctuations in growth conditions, which could arise during growth of multi-layer multi-composition films, eliminated the formation of these complex defects.

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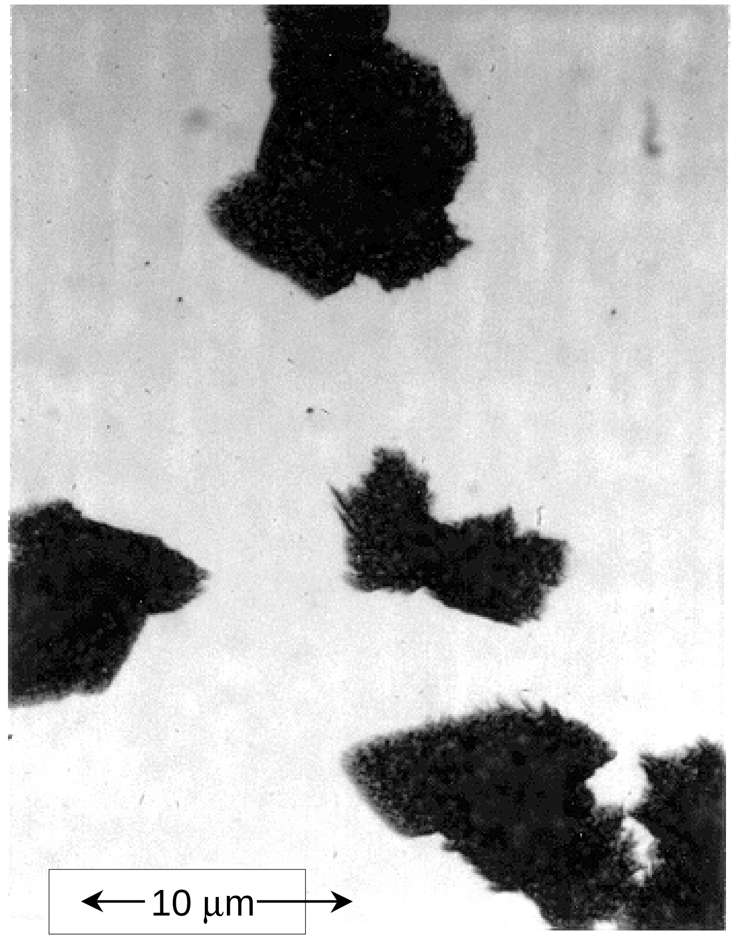

Figure 1

DCA 307

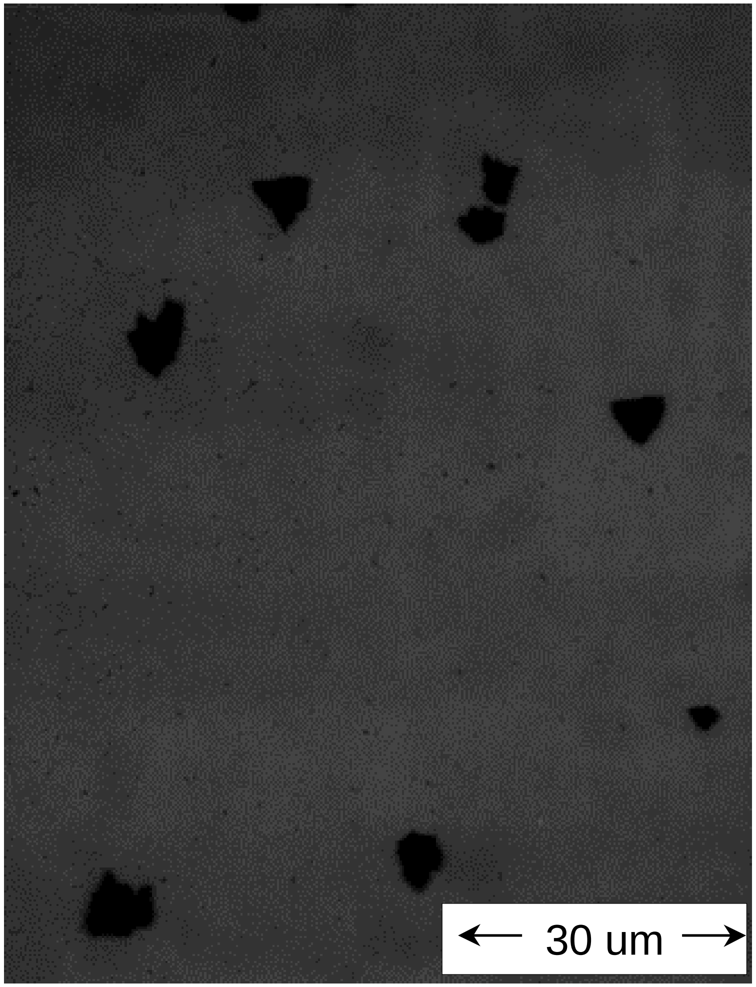

Figure 2

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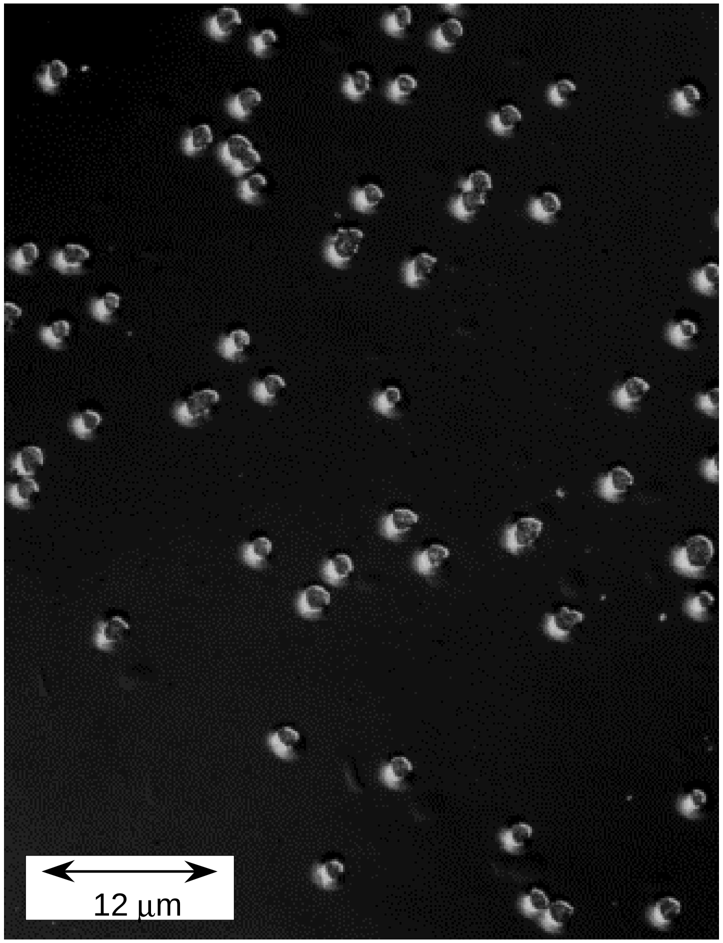

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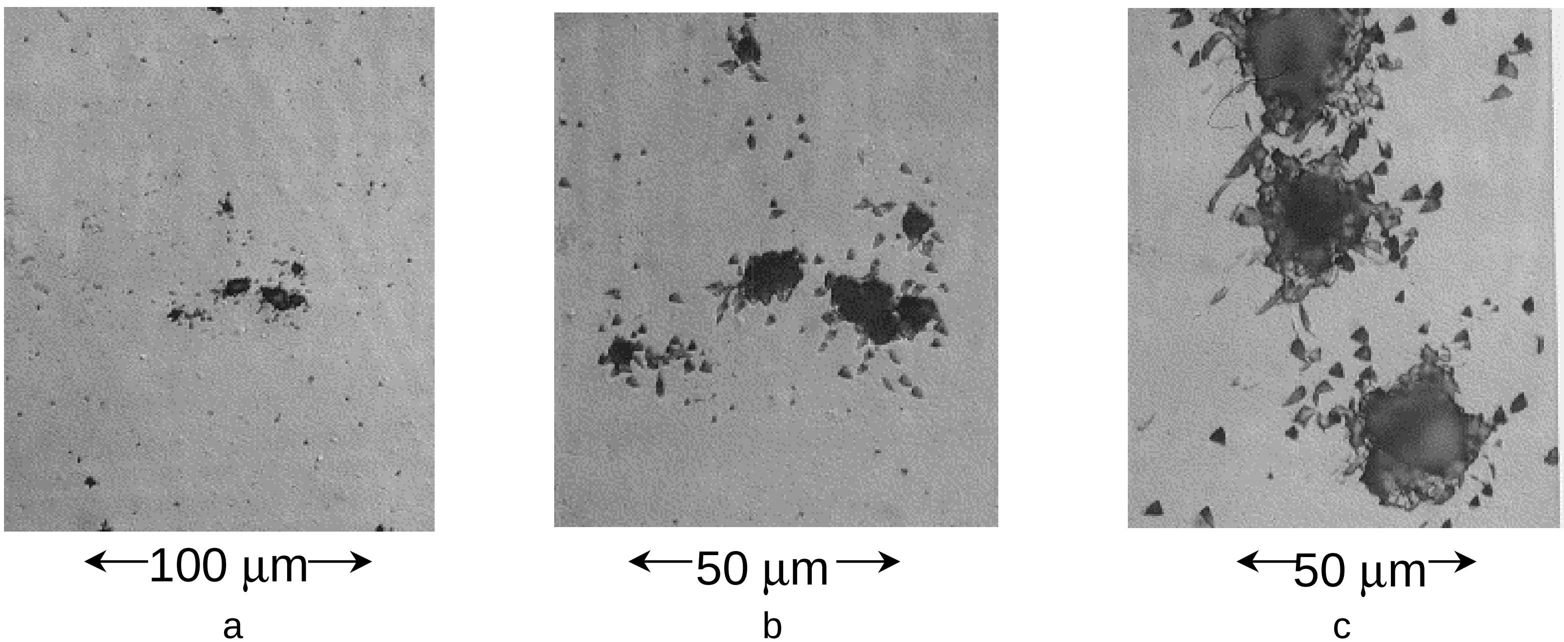

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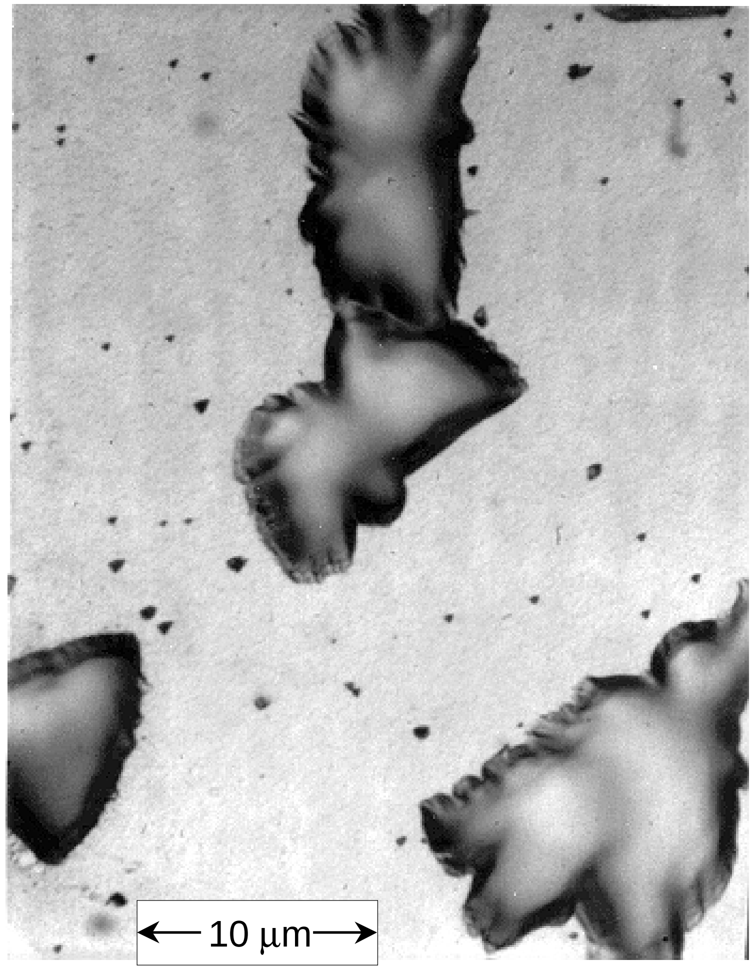

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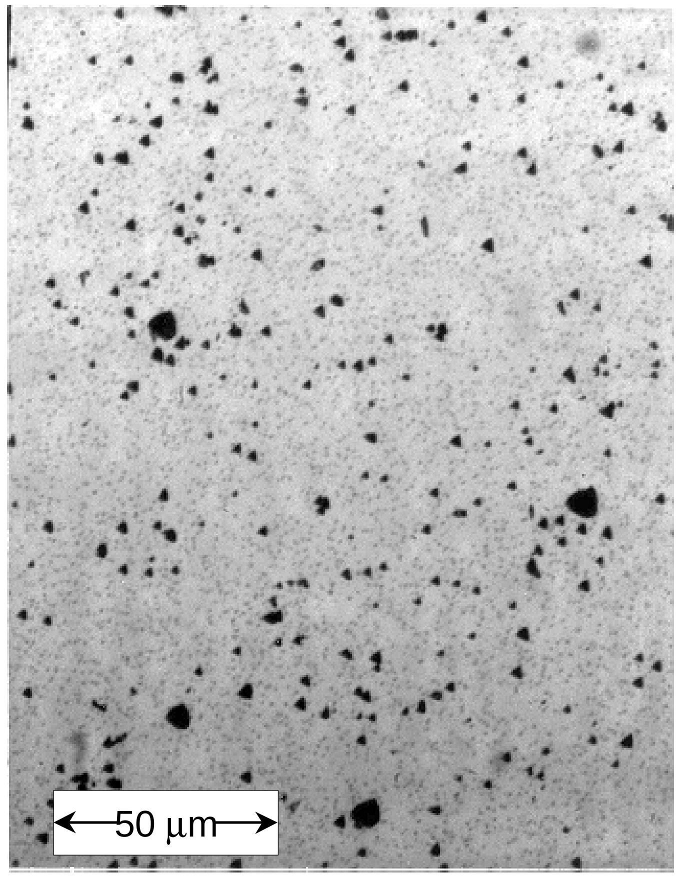


Figure 6

R 1781

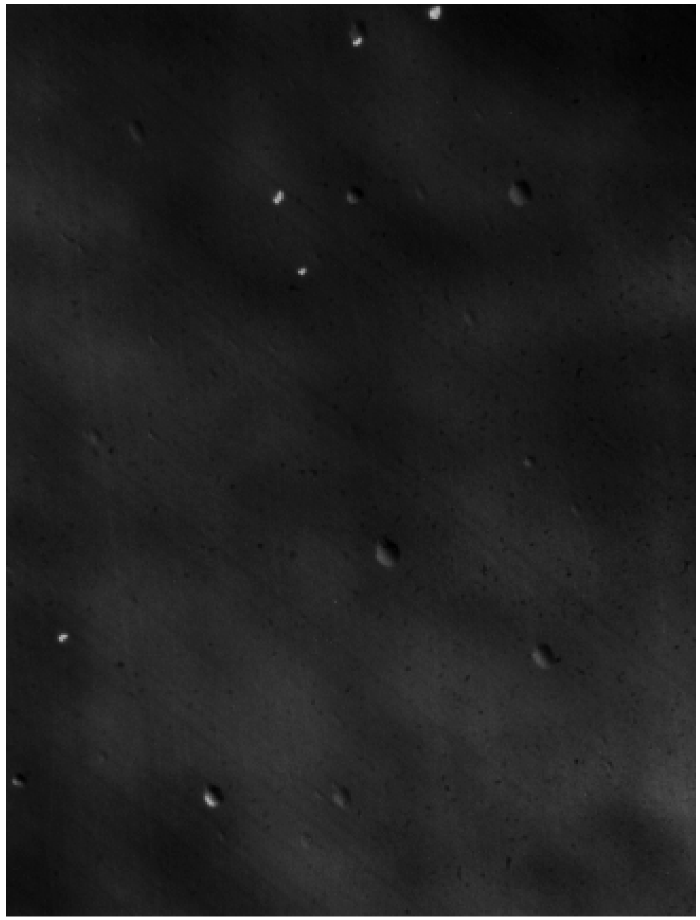
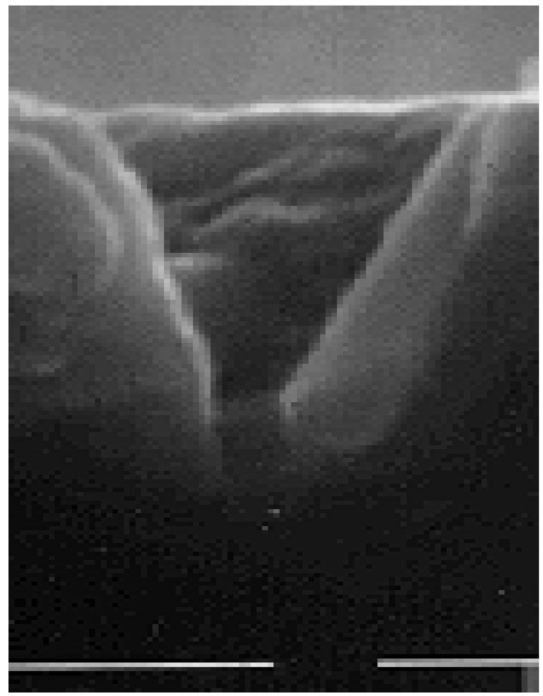


Figure 7

DCA 288



←— 10 μm —→

Figure 8

DCA 334

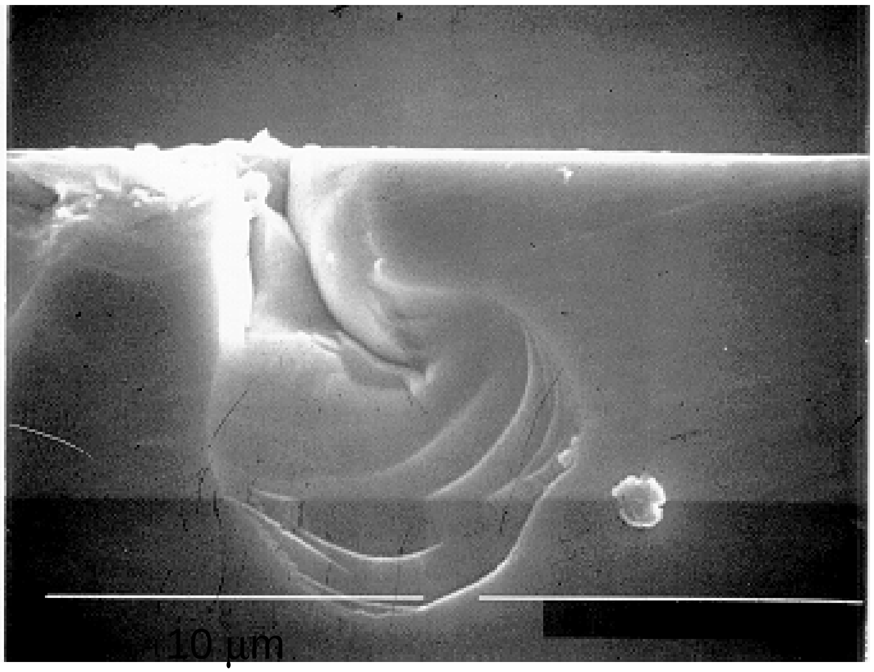
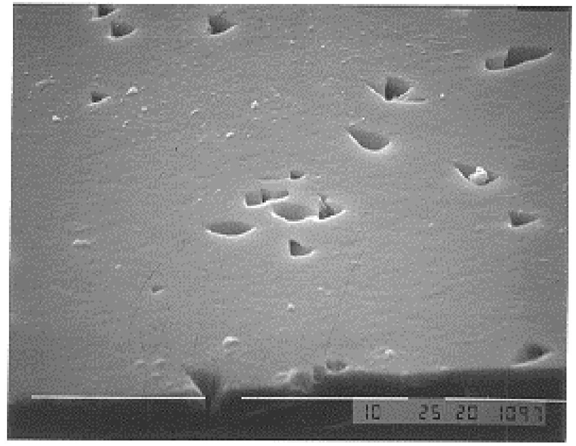
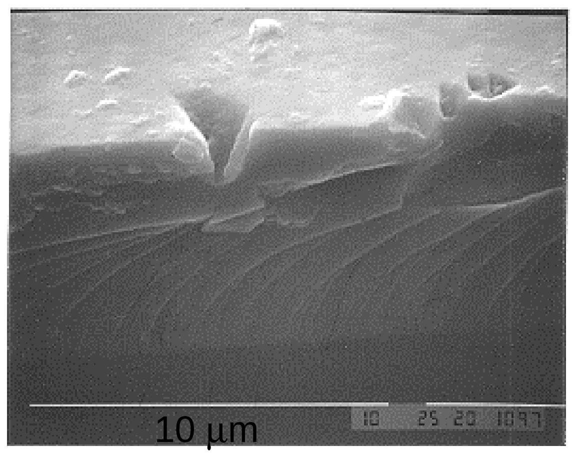


Figure 9

DCA 314

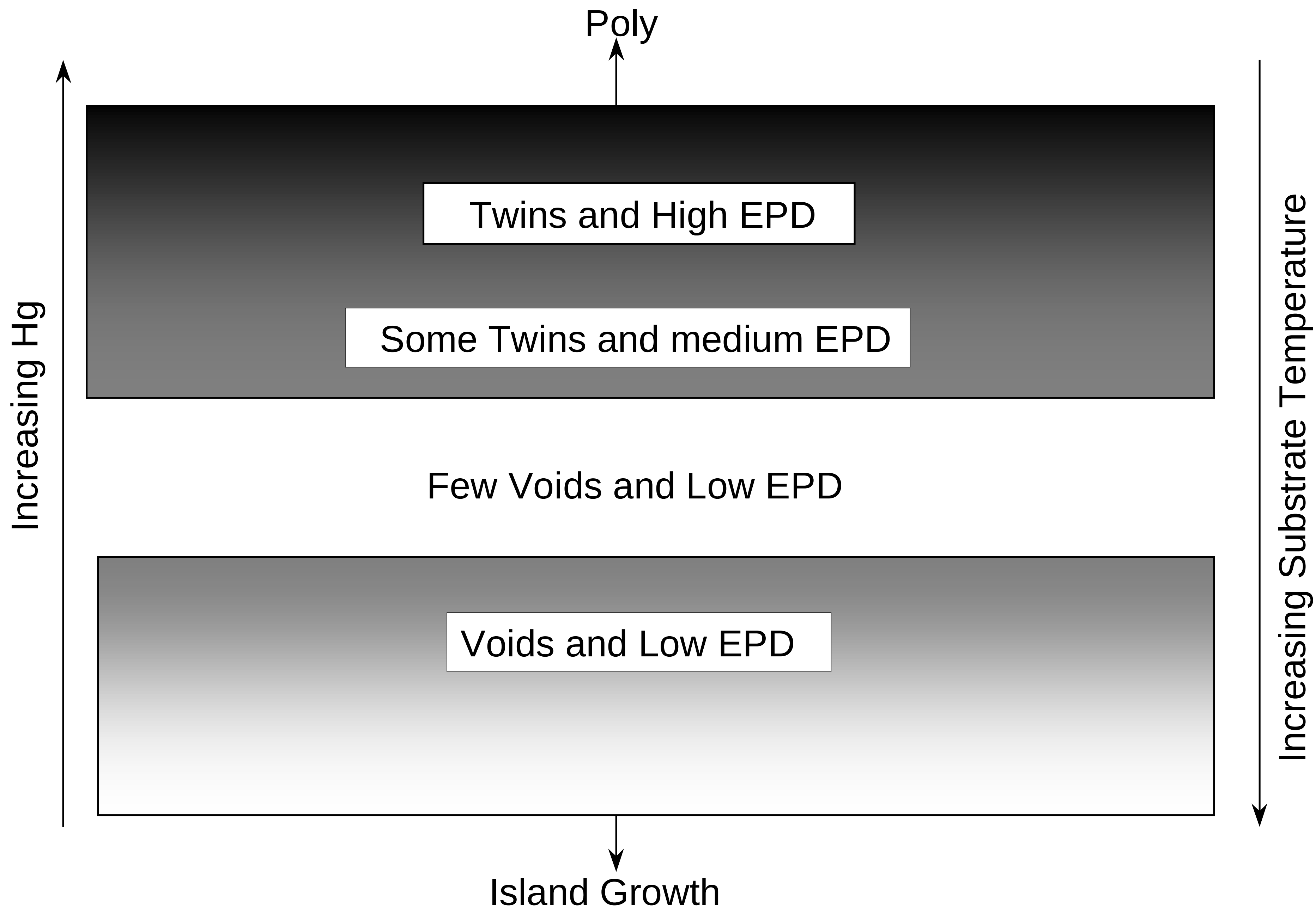


b

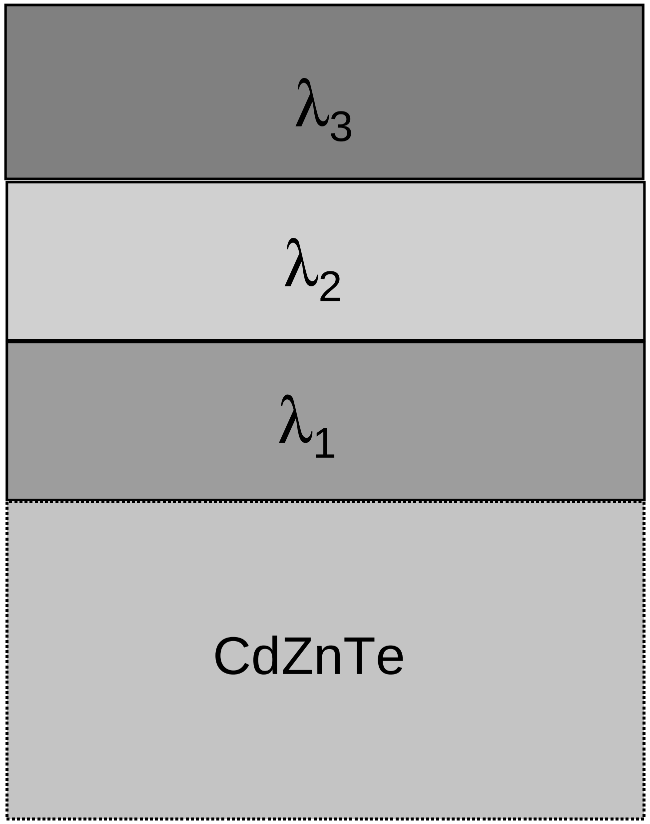


a

Figure 10


Poly
Twins and High EPD
Some Twins and medium EPD
Few Voids and Low EPD
Voids and Low EPD
Island Growth
Increasing Hg
Increasing Substrate Temperature
Figure 11

Schematic Growth of Multi-Layer MBE Film



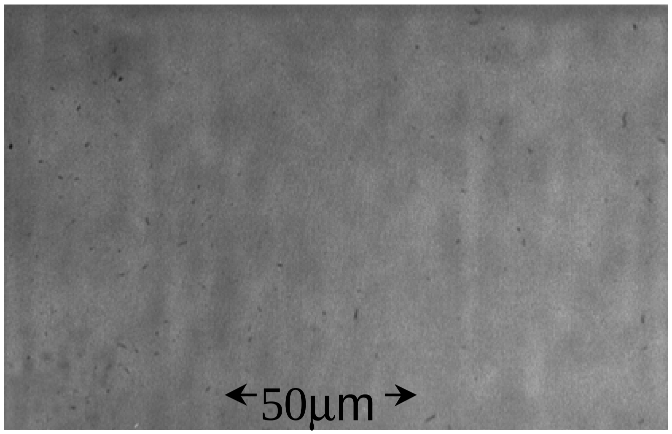
$f\,(T_3\,,\,Hg_3\,,\,CdTe_3\,,\,Te_3\,)$

$f\,(T_2\,,\,Hg_2\,,\,CdTe_2\,,\,Te_2\,)$

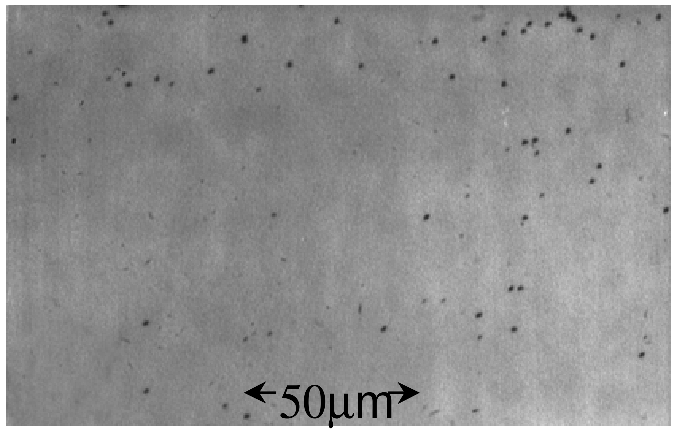
$f\,(T_1\,,\,Hg_1\,,\,CdTe_1\,,\,Te_1\,)$

Figure 12

DCA 397



Void density = 230 cm^{-2}



Dislocation density
= 8.1×10^4 cm^{-2}

Figure 13